- Catalyst Regeneration
  - **Inert Gas Desorption**
    - Ramp up bed temperatures after reduction to 550°C ← **ΔT 50°C above final burn T**
    - Desorb traces of CO, CO_2 and water with high temperature N_2
    - Monitor CO, CO_2 and water in the effluent stream
    - End of desorption signaled by **water concentration below 1 ppmv**
    - It may take between 24 – 72 hrs of desorption to achieve the specification

Catalyst Regeneration

Step	Description	Regeneration Parameters							Spool Piece Location	
		N2 flow kg/h	Air flow kg/h	H2 flow kg/h	FR-2302 COT °C	Catalyst Bed Temp °C	Time hrs	Notes	A	B
1. **Start regen sequence**	- Oper/Regen key switch HS-2301 is used to select R-2301A regeneration - Select Position 2: "A" Regen/"B" Feed to regenerate R-2301A - Select Position 1: "A" Feed/"B" Regen to regenerate R-2301B	-	-	-	Ambient	Ambient or higher			Out	Out
2. **Catalyst bed warm purge**	- Install spool "A" in flare vent line - Lineup N2 through Regen Heater FR-2302, to reactor, to flare - Light FR-2302 main burner, and start increasing catalyst temp from ambient to 400°C, at a rate not exceeding 30°C/hr - Make sure MP Steam to FR-2302 is blinded	Increase to 6,896	-	-	Increase to 420 - 450 or as needed	Increase to 400	12.0 hrs (approx.) for ramping of bed temp	Effluent can be directed to atmosphere when the hydrocarbon in the vent to flare is below 100 ppmv (or local regulation limits).	Flare	Out
3. **Heating**	- Maintain 400°C with hot N2 to flare - Hydrocarbon levels should be below 100 ppmv (or the limit set by local environmental regulations) at end of heating	6,896	-	-	Control at 420 - 450 or as needed	Maintain at 400	4.0 hrs holding time	Step is complete when the hydrocarbon in the vent to flare reaches required limit and the full time has elapsed	Flare	Out
4. **Oxidation and activation #1** (low O2 content)	- Install spool "A" in plant air line - Install spool "B" in the vent to atmosphere - Line up air flow to FR-2302, to reactor, to atm - Start with small flow of air with FC-2351 in manual - Increase air flow to 1,190 kg/h, which is equivalent to 3 vol-% O2 - Ramp up air flow very slowly - monitor exotherm, do not exceed 450°C at any TI in the catalyst bed - Oxidation step is intented for coke removal	6,896	Increase to 1,190 (3 vol-% O2)	-	Increase to 420 - 450 or as needed	Maintain at 400 Do not exceed 450°C	48.0 hrs (approx.) for ramping air 8.0 hrs hold	Step complete when burning appears to have ceased at 400°C (no exotherm)	Air line	Atm vent
5. **Oxidation and activation #2** (higher O2 content)	- Increase temperature to 460°C - Increase air flow to 2,855 kg/h, which is equivalent to 6 vol-% O2 - Oxidation step is intented for coke removal	6,896	Increase to 2,855 (6 vol-% O2)	-	Control at 480 - 510 or as needed	Increase to 460 Do not exceed 485°C	6.0 hrs (approx.) for ramping of bed temp 22.0 hrs (approx.)	Step is complete when the Rx outlet CO2 minus Rx inlet CO2 is <20 ppmv and burning appears to have ceased at 460°C	Air line	Atm vent
6. **Final burn** (with 100% air)	- Increase reactor temp to 500°C, at a rate not exceeding 50°C/hr - Increase air flow to 6,896 kg/h and decrease nitrogen flow to 0 kg/h, which is equivalent to 21 vol% O2 - Final burn step is intended to burn final traces of coke	Decrease to 0	Increase to 6,896 (21 vol-% O2)	-	Control at 520 - 550 or as needed	Increase to 500 Do not exceed 525°C	4.0 hrs (approx.) for ramping of bed temp 12.0 hrs hold	Step is complete when the Rx outlet CO2 minus Rx inlet CO2 is <20 ppmv and burning appears to have ceased at 500°C	Air line	Atm vent



Catalyst Regeneration

Step	Description	Regeneration Parameters							Spool Piece Location	
		N2 flow kg/h	Air flow kg/h	H2 flow kg/h	FR-2302 COT °C	Catalyst Bed Temp °C	Time hrs	Notes	A	B
7. Cooling	- Stop air and increase N2 flow to 6,896 kg/h - Remove spool "A" from plant air line (Regen Heater feed) - Cool reactor bed to 400°C at no more than 50°C/hr	Increase to 6,896	Decrease to 0	-	Decrease to 380 - 410 or as needed	Decrease to 400	10.0 hrs (approx.) for ramping of bed temp	At the end of this step all bed thermocouples should read in the range 390 - 405°C	Out	Atm vent
8. Reduction	- Ensure all reactor bed TI's read 390 - 405°C - Install spool "A" in line to flare - Move spool "B" to hydrogen line (Regen Heater feed) - Start H2 flow to FR-2302, to reactor, to flare - Set H2 flow to 56 kg/h, which is equivalent to 10 vol-% H2 - Perform catalyst reduction for a full 30 minutes period, while making sure that the H2 concentration in the reducing gas is 10 vol-%	6,896	-	Increase to 56 (10 vol-% H2)	Control at 380 - 410 or as needed	Maintain at 400	0.5 hrs hold after reaching specified H2 flow	Start the 0.5-hr timer after reaching the specified H2 flow. Be careful not to exceed 410°C at any bed thermocouple during reduction	Flare	H2 line
9. Purge	- Stop H2 and maintain N2 flow - Purge with N2 to flare until no more H2 in reactor offgas - Remove spool "B" from H2 line	6,896	-	Decrease to 0	Control at 420 - 450 or as needed	Maintain at 400	1.0 hr hold	Hold for at least 1 hr	Flare	Out
10. Desorption	- Install spool "B" in atmospheric vent line - Continue purging with N2 to atmosphere - Close isolation valve in line to flare - Increase reactor bed temp to 550°C at no more than 50°C/hr - Perform inert gas desorption with hot N2 to atmosphere - Monitor the moisture (H2O) in the Rx feed and effluent (verify with portable dew point meter) - Perform desorption until Rx effluent moisture minus Rx feed moisture is ≤ 1 ppmv	6,896	-	-	Increase to 560 - 590 or as needed	Increase to 550	3.0 hrs (approx.) for ramping of inlet temp 48.0 hrs (approx.) hold	Keep all bed thermocouples >540°C. Desorption is complete when delta H2O is ≤1 ppmv. Time is approximate; typically it can take 16 hrs to achieve this delta moisture	Flare (line to flare is isolated)	Atm vent
11. Cooling and hold under N2 pressure	- Cool down reactor to 260°C at no more than 50 °C/hr - Stop N2 flow, shut down and isolate Regen Heater - Pressure purge reactor 3 times from 4.5 to 0.5 barg with N2 to remove all O2 from dead legs. Make sure to keep a positive pressure at all times - Remove spool "B" from atmospheric vent line - Hold reactor under N2 pressure	Decrease to 0	-	-	Decrease to 220 - 240 or as needed	Decrease to 260	6.0 hrs (approx.) for ramping of inlet temp 4.0 hrs (approx.) for purge	Step is complete when all bed thermocouples read in the range 240 - 260°C	Flare (line to flare is isolated)	Atm vent
12. Pressuring	- R-2301A is ready to be brought online - Proceed to slowly pressurize the reactor with vapor feed from OCT Heater FR-2302 - Do not feed hydrocarbons if any thermocouple reads >260°C - Follow the same procedure for R-2301B	-	-	-		260			Flare	Out

- Spool piece arrangement

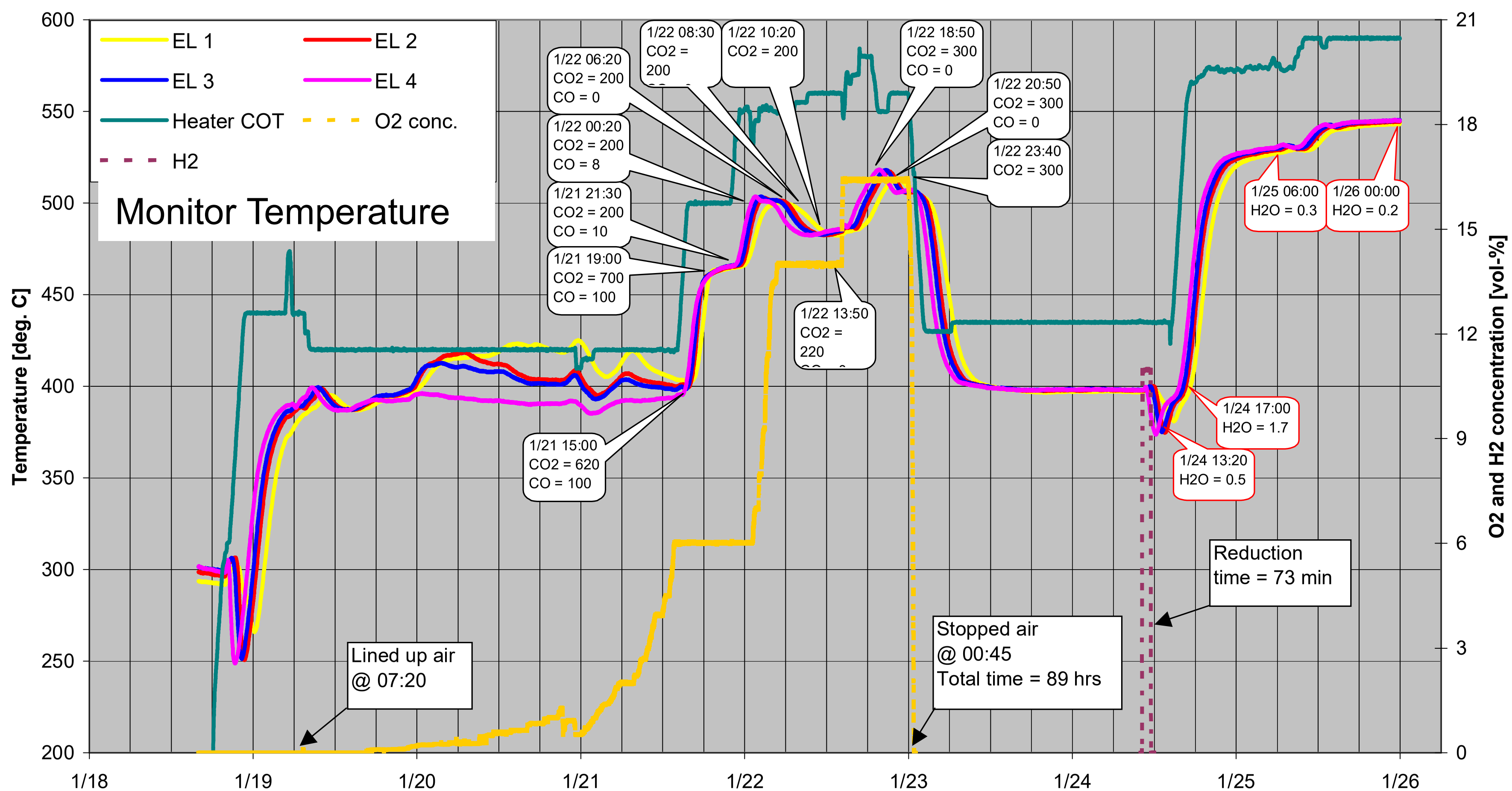


Steps	Location	
	Spool "A"	Spool "B"
Switching	Out	Out
Depressuring	Out	Out
Warm purge	Flare	Out
Heating	Flare	Out
Oxidation	Air Line	Atmospheric Vent
Final Burn	Air Line	Atmospheric Vent
Cooling	Out	Atmospheric Vent
Reduction	Flare	H_2 Line
Purge	Flare	Out
Inert Gas Desorption	Flare (Line to flare is isolated)	Atmospheric Vent
Cooling	Flare (Line to flare is isolated)	Atmospheric Vent (initial)
Repressure	Flare	Out

Catalyst Regeneration – Partial

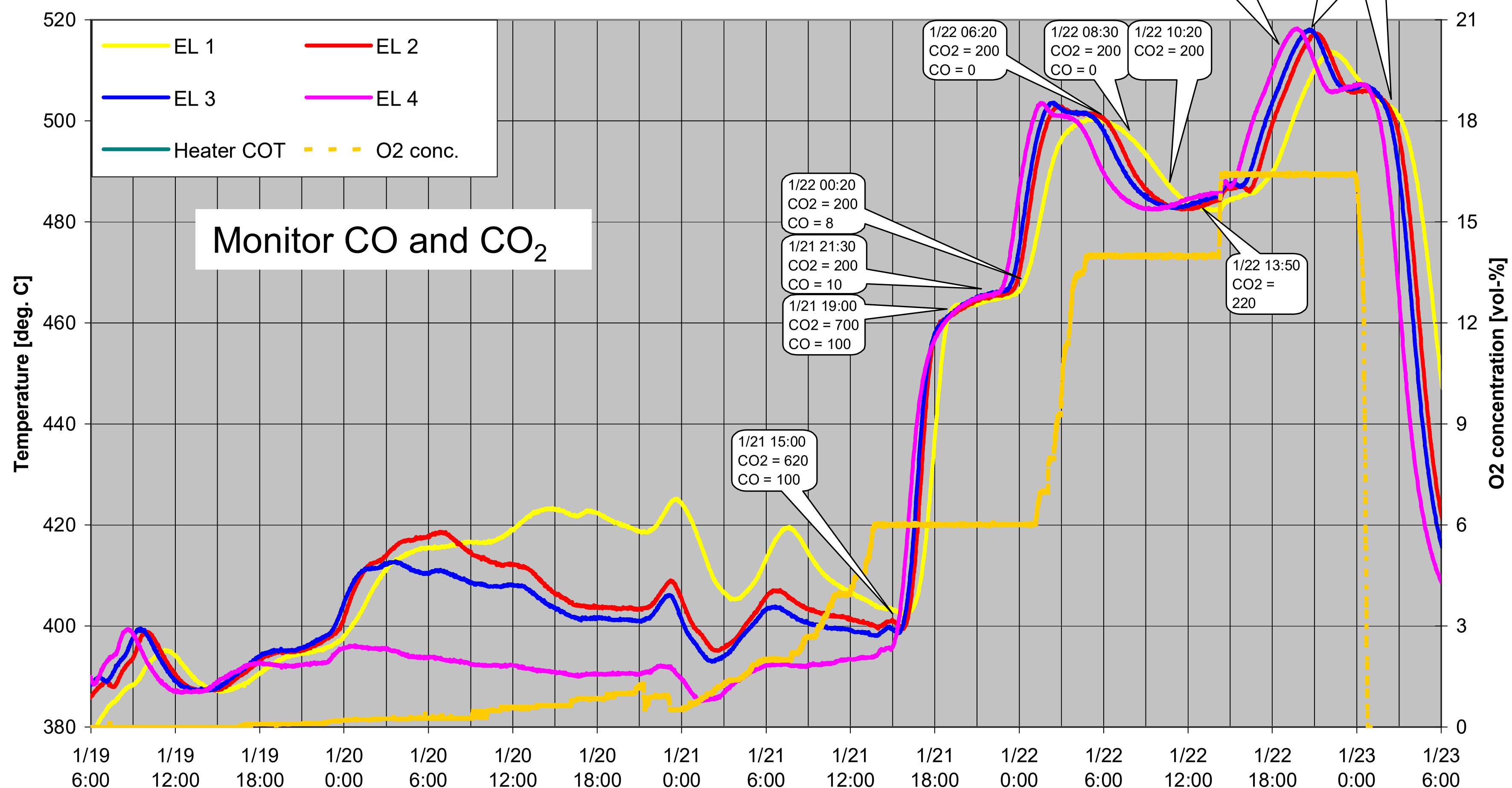
- Generally OCT Reactor will have a significant idle time
- Commercial experience shows that leaving the reactor under N2 blanket for extended period of time, it may not show a high catalyst activity when brought in normal service
- **Recommendation**
  - Stop the regeneration protocol at step 7 (cooling)
    - Cool the reactor down to ambient temperature after Final burn and leave it under positive N2 pressure
  - About 4 days prior to lining up the idle OCT reactor in normal service, make arrangements to do the balance reduction and Inert gas desorption steps
    - Heat up the reactor to reduction temperature
    - Do reduction step 8
    - Followed by purge
    - Do Inert gas gas desorption step 10
    - Cool the reactor to 260 °C, the line-up temperature

Catalyst Regeneration



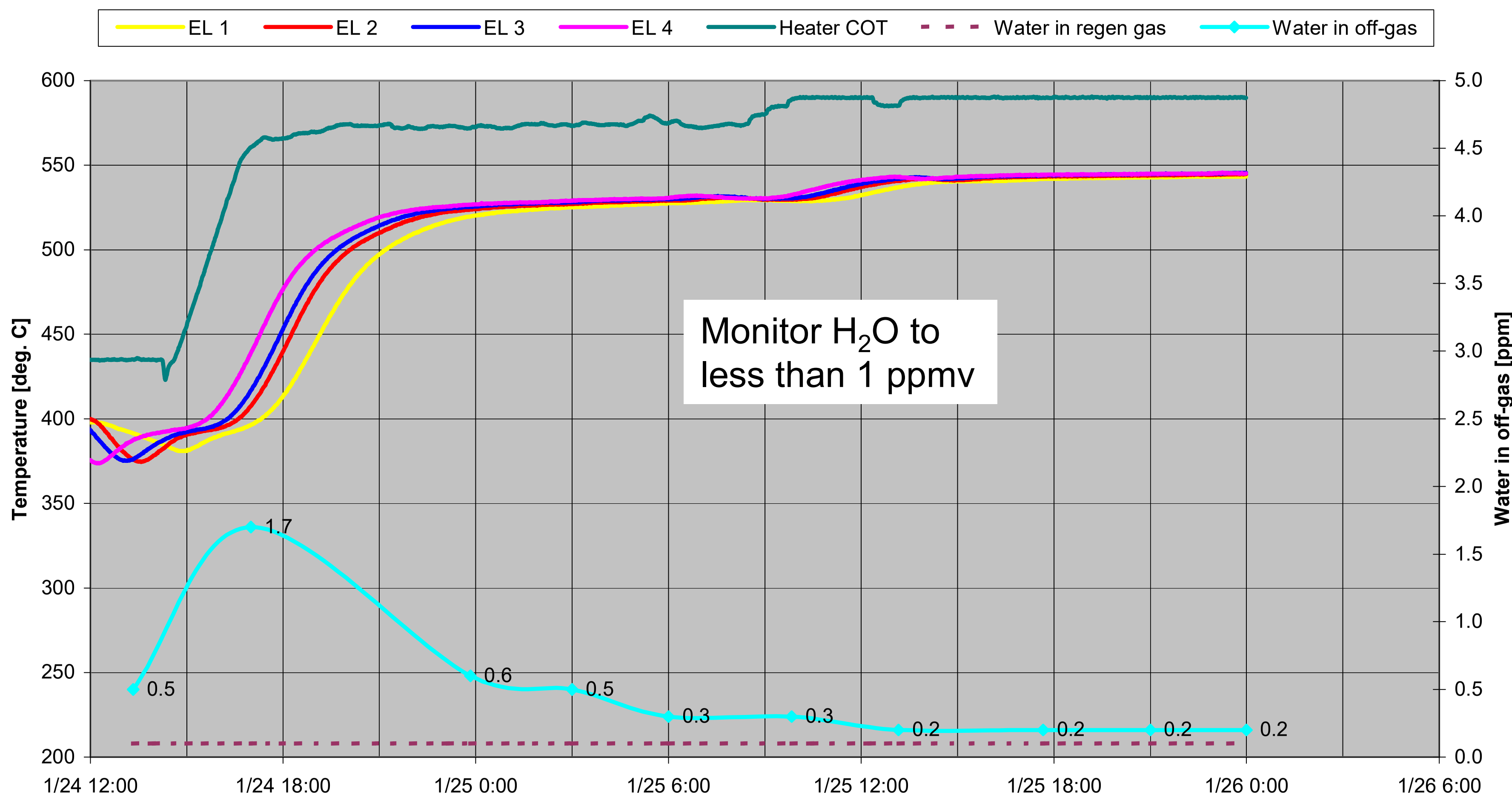
For Reference Only

Catalyst Regeneration



For Reference Only

Catalyst Regeneration - Desorption



For Reference Only

Special Procedures

- Catalyst Activation
  - The OCT Reactor needs to be activated before startup
  - The purpose of activation is to:
    - To remove moisture from Magnesium oxide catalyst

      $Mg(OH)_2 \longrightarrow MgO + H_2O$
    - To bring Tungsten catalyst (WO_3) to active oxidation state
  - The **activation procedure** is very **similar** to the **regeneration procedure**. However, there is no coke to burn

- Catalyst Activation
  - Activation consists of:
    - Initial heating → 350 ^oC Ramp rate 30^oC/h
    - Dryout → 60 hrs @ 350^oC
    - Heating → 460^oC Ramp rate 50^oC/h
    - Oxidation → 8 hrs @ 460^oC
    - Cooling → to 400^oC
    - Reduction with H_2 → 0.5 hrs @ 400^oC with 10% mol-% H_2
    - N_2 Purge → 1 hr @ 400^oC
    - Inert gas desorption → 24-72 hrs & H_2O < 1 ppmv @ 550^oC
    - Cooling and hold under N_2 → To 260^oC

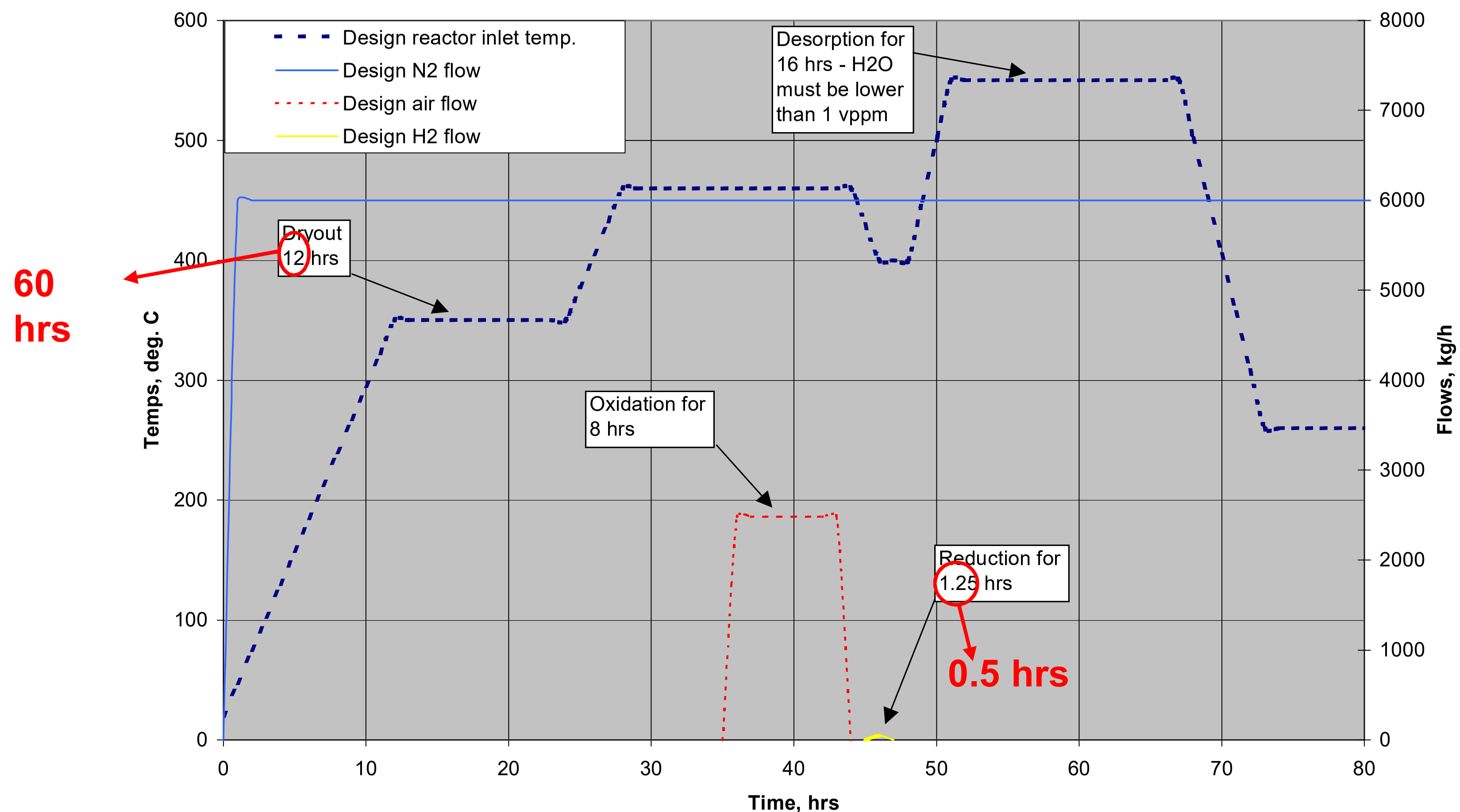
Catalyst Activation

Step	Description	Regeneration Parameters							Spool Piece Location	
		N2 flow kg/h	Air flow kg/h	H2 flow kg/h	FR-2302 COT °C	Catalyst Bed Temp °C	Time hrs	Notes	A	B
1. **Start regen sequence**	- Oper/Regen key switch HS-2301 is used to select R-2301A activation - Select Position 2: "A" Regen/"B" Feed to regenerate R-2301A - Select Position 1: "A" Feed/"B" Regen to regenerate R-2301B	-	-	-	Ambient	Ambient			Out	Out
2. **Catalyst bed initial heating**	- Install spool "B" in atmospheric vent line - Lineup N2 through Regen Heater FR-2302 reactor, to atm - Light FR-2302 main burner, and start increasing catalyst temp from ambient to 320°C, at a rate not exceeding 30°C/hr - Make sure LP Steam to FR-2302 is blinded	Increase to 6,896	-	-	Increase to 340 - 370 or as needed	Increase to 320	24.0 hrs (approx.) for ramping of bed temp	Large delta T is expected to be observed across the catalyst bed during the initial heating and dryout steps. Step is complete when all bed thermocouples read ≥320°C	Out	Atm vent
3. **Dryout**	- Increase catalyst temp to 350°C - Maintain 350°C for 60 hrs (minimum) with hot N2 to atmosphere	6,896	-	-	Control at 370 - 400 or as needed	Maintain at 350	60.0 hrs (minimum) holding time	As catalyst dryout progresses (water is removed), bed delta T will decrease. Allowable temperature range: 350 - 370°C	Out	Atm vent
4. **Heating to oxidation temperature and hold**	- Increase catalyst temp to 460°C, at a rate not exceeding 50°C/hr - Maintain 460°C for 8 hrs with hot N2 to atmosphere	6,896	-	-	Increase to 480 - 510 or as needed	Increase to 460	8.0 hrs (approx.) for ramping of bed temp 8.0 hrs hold	Keep all bed thermocouples reading in the range 450 - 500°C for a minimum of 8 hours	Out	Atm vent
5. **Oxidation and activation**	- Install spool "A" in plant air line (Regen Heater feed) - Start air flow to FR-2302, to reactor, to atm - Increase air flow to 2855 kg/h, which is equivalent to 6 vol-% O2 - Keep catalyst temperature at 460°C - Oxidation step is intented for oxidizing WO3 catalyst to its highest valance state	6,896	Increase to 2,855 (6 vol-% O2)	-	Control at 480 - 510 or as needed	Maintain at 460	1.0 hr to ramp up air flow 8.0 hrs hold	Start the 8-hr timer after reaching specified air flow and all bed thermocouples read in the range 450 - 500°C	Air line	Atm vent
6. **Final burn**	- This step is not required, since there is no coke on the catalyst									

Catalyst Activation

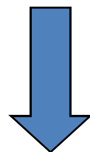
Step	Description	Regeneration Parameters							Spool Piece Location	
		N2 flow kg/h	Air flow kg/h	H2 flow kg/h	FR-2302 COT °C	Catalyst Bed Temp °C	Time hrs	Notes	A	B
7. Cooling	- Stop air and maintain N_2 flow - Remove spool "A" from plant air line (Regen Heater feed) - Cool reactor bed to 400°C at no more than 50°C/hr	6,896	Decrease to 0	-	Decrease to 380 - 410 or as needed	Decrease to 400	8.0 hrs (approx.) for ramping of bed temp	At the end of this step all bed thermocouples should read in the range 390 - 405°C	Out	Atm vent
8. Reduction	- Ensure all reactor bed TI's read 390 - 405°C - Install spool "A" in line to flare - Move spool "B" to hydrogen line (Regen Heater feed) - Start H_2 flow to FR-2302, to reactor, to flare - Set H_2 flow to 56 kg/h, which is equivalent to 10 vol-% H_2 - Perform catalyst reduction for a full 30 minutes period, while making sure that the H_2 concentration in the reducing gas is 10 vol-%	6,896	-	Increase to 56 (10 vol-% H2)	Control at 380 - 410 or as needed	Maintain at 390 - 405	0.5 hrs hold after reaching specified H2 flow	Start the 0.5-hr timer after reaching the specified H2 flow. Be careful not to exceed 410°C at any bed thermocouple during reduction	Flare	H2 line
9. Purge	- Stop H_2 and maintain N_2 flow - Purge with N_2 to flare until no more H_2 in reactor offgas - Remove spool "B" from H_2 line	6,896	-	Decrease to 0	Control at 420 - 450 or as needed	Maintain at 400	1.0 hr hold	Hold for at least 1 hr	Flare	Out
10. Desorption	- Install spool "B" in atmospheric vent line - Continue purging with N_2 to atmosphere - Close isolation valve in line to flare - Increase reactor bed temp to 550°C at no more than 50°C/hr - Perform inert gas desorption with hot N_2 to atmosphere - Monitor the moisture (H_2O) in the Rx feed and effluent (verify with portable dew point meter) - Perform desorption until Rx effluent moisture minus Rx feed moisture is ≤ 1 ppmv	6,896	-	-	Increase to 560 - 590 or as needed	Increase to 550	3.0 hrs (approx.) for ramping of inlet temp 48.0 hrs (approx.) hold	Keep all bed thermocouples >540°C. Desorption is complete when delta H2O is ≤1 ppmv. Time is approximate; typically it can take 24 - 72 hrs to achieve this delta moisture	Flare (line to flare is isolated)	Atm vent
11. Cooling and hold under N2 pressure	- Cool down reactor to 260°C at no more than 50 °C/hr - Stop N_2 flow, shut down and isolate Regen Heater - Pressure purge reactor 3 times from 4.5 to 0.5 barg with N_2 to remove all O_2 from dead legs. Make sure to keep a positive pressure at all times - Remove spool "B" from atmospheric vent line - Hold reactor under N_2 pressure	Decrease to 0	-	-	Decrease to 220 - 240 or as needed	Decrease to 260	6.0 hrs (approx.) for ramping of inlet temp 4.0 hrs (approx.) for purge	Step is complete when all bed thermocouples read in the range 240 - 260°C	Flare (line to flare is isolated)	Atm vent
12. Pressuring	- R-2301A is ready to be brought online - Proceed to slowly pressurize the reactor with vapor C4's from OCT Heater FR-2301 once the OCU loop is in operation - Do not feed hydrocarbons if any thermocouple reads >260°C - Follow the same procedure for R-2301B	-	-	-		260			Flare	Out

Catalyst Activation



For Reference

- OCT Reactor Switching
  1. Reduce the outlet temperature of the OCT Reactor Feed Heater to 304 °C. Wait for Reactor inlet temperature to reach 304°C and maximum 311 °C at outlet.

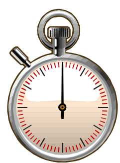


at bed thermocouples of Regenerated Reactor T≤260°C

at inlet temperature of normal operating Reactor T≤304°C

If **ok**, proceed to next purge and pressurize step

- **OCT Reactor Switching**
  2. Pressurize and purge the standby reactor with hydrocarbon
     - Reactor lined-up to flare
     - Pressure to 10 kg/cm2g and depressure to remove nitrogen
     - Pressure purge 3 times.
  3. Pressurize with hydrocarbon to normal operating pressure 30.2 kg/cm^2g
  4. Open standby reactor outlet and inlet valves. Reactors are now in parallel operation.



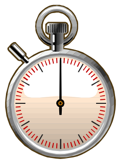
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or

Bed T>320°C → **Depressurize the Reactor**

Special Procedures

- OCT Reactor Switching
  5. Isolate and Depressure of Operating reactor
  6. Isolate operating reactor
  7. Depressure the operating reactor



< 1hr


CB&I



RABIGH REFINING AND PETROCHEMICAL COMPANY (ARAMCO OVERSEAS COMPANY B.V. & SUMITOMO CHEMICAL CO., LTD.)

TECHNOLOGY TRAINING

PART G

FIELD EXPERIENCES





Plants in Operation

Issues

- 1. Safety
  - Exotherms in OCT Reactors
  - Tube rupture in OCT Heaters
- 2. Design
  - Inadequate venting (purge) capacity in OCU
  - Feed stock analysis
  - Actual plant modifications
- 3. Catalyst
  - Proper selection of support balls
  - High pressure drop
  - Loading density
  - OCT reactor screen mesh and wire sizes
  - Consequences of catalyst poison in feed

Issues

- 4. Treater Design and Operation
  - Impurity Breakthrough
  - Regeneration
  - Instrumentation
  - Oligomers
  - Other
- 5. Olefins Conversion Unit

CBI

Issues

- 6. Engineering
  - Hot Vapor bypass
  - CW velocity too low
  - Heat losses

- 7. Construction
  - Inadequate insulation on regeneration lines
  - Improper location of temperature controllers
  - Cryogenic Installations
  - Electric Heater
  - Butene Stripping Vaporizer

Issues

- 8. Commissioning
  - Construction debris
  - Cleaning
- 9. Laboratory
  - Analytical
  - Analyzer/Sample Piping
- 10. Operations

- OCT Reactor Exotherm - Problem
  - High temperature exotherm occurred at one of our units – temperatures as high as 650°C were registered by bed thermocouples
    - Exotherm occurred during reactor switch
    - Reactor was not fully cooled after regeneration – some bed thermocouples registered 340°C - 410°C
    - Reactor was under no-flow condition for 30 minutes
    - Very high reaction rate above 365°C
  - Mild exotherms (<15 °C) seen at two plants
    - Exotherms occurred when on-line reactors tripped
    - Reactors held at pressure and temperature to extended periods (30 min to 2.5 hour)
    - Exotherms subsided when flow established

- OCT Reactor Exotherm - Analysis
  - Exotherms occur due to over-reduction of WO_3 catalyst
  - Oxygen released can oxidize hydrocarbons liberating heat
  - Ethylene decomposition is unlikely but may have occurred
  - There is insufficient oxygen in the catalyst to uniformly raise the catalyst bed and reactor to above 370°C
  - Localized heating most likely responsible for high thermocouple readings

- OCT Reactor Exotherm - Solution
  - New plant design incorporates additional features to prevent occurrence of exotherms:
    - Revised OCT Reactor switching procedure emphasizes need to switch at proper temperatures (260°C for fresh reactor/304°C for used reactor) and time limits given
    - Maximum operating temperature reduced from 370°C to 340°C
    - High-High Temperature interlock set-point reduced to 350°C which provides adequate time for operator action, if necessary
    - High-High Temperature interlock incorporates individual reactor depressuring
    - OCT Heater and OCT Reactor interlocks cascaded to prevent no-flow situation in reactor
    - Run-mode and pressure switches reset interlock temperature at switching/start-up

- OCT Heater Tube Rupture - Problem
  - Tube rupture occurred during start-up at two units
  - Overfiring during C4 liquid circulation caused excessively high cross-over temperature leading to tube rupture in radiant section
  - C4 circulation rate was excessively high (160 – 300%)
  - Operators ignored temperature alarms
  - Vaporization duty was higher than design duty of heater
  - Maldistribution of flow in heater is suspected

- OCT Heater Tube Rupture - Solution
  - New plant design incorporates additional features to prevent occurrence of tube rupture:
    - Separate start-up vaporizer (steam) is provided – OCT Heater does not need to provide vaporization duty
    - FI’s installed on all heater passes to monitor flow distribution at start-up
    - TI’s installed on heater crossover to monitor temperature during start-up
    - Revised low-pressure start-up method to insure complete vaporization at heater inlet and good flow distribution among the passes

- Inadequate Venting Capacity – Problem
  - Deethylenizer vent sized for normal inert levels (H_2, CH_4) in ethylene feed and C_4 feed
  - N_2 blanketing of C4 Surge drum introduces additional inerts
  - Feedstocks may contain higher than design inert levels
  - N_2 blanketing creates high inert levels at start-up

- Inadequate Venting Capacity – Solution
  - Vent capacity increased to allow for abnormal conditions
  - Normal flow rate increased to 3% of ethylene feed
  - Vent valve sizing basis increased (30% open at design rate)
  - N_2 padding minimized/eliminated from C_4 Surge Drums
  - Start-up procedure modified to allow adequate purging of N_2 from system

Design

Feedstock analysis and review of feed treatment should be performed at multiple points during project



At proposal stage – correct flow sheet

Before BEP kick off – correct basis

Before detailed engineering is complete – tie-ins, confirm basis

Before start-up – modifications to operation

After start-up – trouble shooting

Lummus Technology can design a plant to process any feed, **but impurities need to be known**

Actual Plant Modifications

Impurity	Impact	Removal
MTBE	Shortens OCT reactor run length	New distillation column
Green Oil	Shortens OCT reactor run length Fouls OCT reactor feed heating Damages analyzers	New distillation column
High Boiling Compounds	Accumulates in reboilers Fouls OCT reactor feed heating Damages analyzers	Modify upstream unit
Inhibitors (e.g., TBC, BHT)	Irreversibly adsorbs to treaters; damages them in a matter of days Shortens OCT reactor run length	Modify upstream unit New distillation column New guard beds

Successful start-up depends good feed testing



Catalyst

- OCT Catalyst Support Balls - Problem
  - Support balls at one of our units disintegrated creating fines
  - Fines created high pressure drop and lifted bed
  - Support balls and catalyst were crushed

- OCT Catalyst Support Balls - Solution
  - Support balls were found to have high calcium content
  - Only high alumina content balls are now specified (Denstone 99 or equivalent)

OCT Reactor Screen Mesh and Wire Sizes

- Catalyst / Adsorbent support screens need to be:
  - Strong enough to support the catalyst charge
  - Small enough gap so that catalyst / adsorbent does not drop through
  - Sufficient open area: avoid Δp issues

- The exact dimensions can vary based on the detailed reactor construction – check with Lummus.
- An extreme example of a screen problem encountered in the field is illustrated on the next slide.



Lummus Technology BEP support screen configuration

Mesh size	Wire diameter	Open Area (%)
10	0.38mm	73
4	0.91mm	73

OCT Reactor Screen Mesh and Wire Sizes

Example

- Initial screens made by the engineering firm:
  - 4 mesh with 3.0 mm wire diameter: 28% open area
  - 20 mesh with 0.408 mm diameter: 46% open area
  - Extra screens added between catalyst and ceramic balls

- After on site review and discussion with Lummus, screens changed to:
  - 4 mesh with 0.91 mm diameter: 73% open area
  - Removed all of the 20 mesh screens
  - Removed extra screens

- The Same Nominal Mesh Size Can Have a Large Difference in Open Area
- US Mesh size is defined as “the number of openings per linear inch”
  - 4 mesh has 4 openings per inch

Mesh Size	American Wire Gage (AWG)	Standard Wire Gauge (SWG)	Wire Size (mm)	Width of Opening (mm)	Open Area (%)
4 Mesh	9	11	2.9	3.4	29
	13	15	1.8	4.5	51
	15	17	1.45	4.9	60
	18	19	1.02	5.3	70
Lummus recommended	19	20	0.91	5.4	73
	20	21	0.81	5.5	76

X-066 Catalyst Screening

Hand screening is the most effective way to remove the small amounts of fines generated during transportation

Problems Observed:

- Incorrect screen used, so too much or too little fines removed
- **Correct screen is 12 mesh with 0.8mm wire diameter (removes particles <1.3mm)**
- Mechanical screening used created 7-8% fines





OCT Catalyst Loading

Careful, efficient loading is an investment in stable reactor performance and catalyst life.

Problems Observed:

- Screened catalyst bags stacked 2 high **(recommend single height)**
  - Excess handling of stacking process can cause crushing
- Big bag support on loading hopper missing - Safety issue!
- Initial fill of hopper – free fall too high **(Recommend ≤ 0.5m)**
- Loading sock ≥ 6” (15 cm) diameter
  - Catalyst free falling leading to breakage and fines
  - <u>**4” (10cm) diameter fabric sock is correct**</u>
- Bed leveling missed
  - Inconsistent packing density
  - Non-uniform reactant flow through the catalyst bed

Feed Impurities Removal

Problems Observed:

- Higher impurity content of feed than original treater design specifications:
  - Impact: Insufficient adsorbent capacity (reduced OCT cycle-length)
- Extended treater cycle-lengths, beyond 48hr design, allowing contaminant breakthrough to OCT reactor:
  - Impact: Reduced OCT cycle length & may accelerate OCT catalyst deactivation
- Unable to reach optimum treater regeneration temperature (290°C)
  - Impact: Incomplete desorption, lower treater capacity
- Unable to apply sufficient gas flow rate
  - Impact: Extends treater desorption time required

- OCT Catalyst High Pressure Drop - Problem
  - Some plants experienced high pressure drop during normal operation and regeneration
    - Causes
      - Improper loading - crushing the catalyst
      - Inferior support balls
  - High pressure drop lead to maldistribution and poor C_4 conversion
  - Bed lifting may have occurred during regeneration leading to crushed catalyst

- OCT Catalyst High Pressure Drop – Problem
  - High pressure drop
    - Crushed inert balls in OCT Reactor, cause for high pressure drop:



- OCT Catalyst High Pressure Drop - Solution
  - Catalyst production method was changed to provide catalyst with lower fines content when shipped
  - Catalyst is now screened before loading at site
  - Catalyst loading procedure modified to prevent fines formation during loading
  - Support grid now specified with higher loading to allow operation at higher pressure drop if necessary
  - Regeneration guidelines provided to avoid bed lifting

- OCT Catalyst Loading Density - Problem
  - Loading of X-052 and X-066 catalyst must be done by weight
  - In some reactors available bed volume not enough to allow 100% loading of catalyst by weight
    - Actual loading density of catalyst lower than ABD (design average bulk density)
    - Volume occupied by reactor thermowells not taken into account – it can be as high as 2-5% of total bed volume

- OCT Catalyst Loading Density - Solution
  - In new designs, Lummus is accounting for 10% lower packing density
  - In reactors already built, recommendation is to load up design weight of X-066, at the expense of loading less-than-design X-052 weight
  - Clients should submit as-built drawings of reactors not loaded up yet to Lummus in order to determine whether additional space can be allocated in the vessel

CBI

Consequences of Catalyst Poisons in Feed

- Several plants have had to make modifications after start-up due to unknown feedstock poisons
- Modifications have included:
  - Installation of new distillation column $$$
  - Installation of new feed treaters $$
  - Replacement of reactor catalyst $$
  - Replacement of treater adsorbent $
  - Change analyzer sample systems to cope with high boilers $



Treater Design and Operation

- Treater Design and Operation – Problems
  - Breakthrough of impurities at operating units leads to reduced C4 conversion and shorter cycle time
    - Feedstocks contain higher than design impurity levels or alternate impurities not accounted for in original design
    - Regeneration conditions (time, temperature) not met
    - Reactivity of some components (e.g. butadiene, isobutene) can reduce treater effectiveness
    - Polymerization inhibitor present in feedstock
    - C4 left on adsorbent after draining may cause coking
    - Poor level indication when draining and filling

Treater Design and Operation

- Treater Design and Operation – Solutions
  - Always include treating within OCU reactor loop even if fresh feeds are treated
  - Regeneration at 270 to 280°C minimum – no more low temperature regeneration
  - Time-temperature regeneration profile strictly adhered to by client
  - Where necessary, additional pretreatment installed (e.g. DME removal, MTBE removal)
  - BHT not allowed in feedstock; TBC removed
  - Proper selection of adsorbents where reactive components exist – BASF's Selexsorb CDL or UOP's AZ300



Treater Design and Operation

- Treater Design and Operation – Solutions
  - C4 treater regeneration cycle modified to include C4 stripping to maximize C4 removal and recovery after draining
  - Level instruments changed to diaphragm type to eliminate fouling from adsorbent fines
  - Butene stripping liquid can build-up in the bottom. Newer designs have butene warming gas leaving from the bottom head
  - Fill lines to drain to expedite filling process and minimize perturbation in the forward flow

Treater Design and Operation

- Other problems
  - Poor level indication in Treaters:
    - Wrong installation of LT’s
    - Wrong calibration of LT’s
    - Wrong filling fluid
  - Excessive plugging of filter elements
  - Installation of filter with smaller-than-design mesh
  - Water and TBA breakthrough from Treaters due to upsets in upstream units, lack of communication between units
  - Wrong set-points of Treater interlocks
  - Excessive leaking of Treaters’ XV’s
  - Liquid-filling Treaters too slowly → high exotherm
  - Putting jumpers in Treaters interlocks



Treater Design and Operation

- During startup, when preload system is not available…
  - Make sure to fill up Treaters with liquid quickly, and to keep the pressure in the Treater during filling low and under control
    - If not, very high exotherms could occur leading to premature fouling of the bed
    - If possible, it is recommended to fill up Drain Drum first, and then quickly transfer inventory to Treater

Olefins Conversion Unit

- OCT Reactor
  - Improperly activating OCT Reactor catalyst
  - Difficulty in controlling regen air flow:
    - Size of air valves
    - Range of FE
    - Distance between regen heater and reactors
  - Water analyzer in regen line installed in wrong location
  - High concentration of contaminants in OCU feed → sampling required!
  - Not properly purging HC with N_2 from OCT Reactor after shutdown
  - Operating with too low E/B ratio



Olefins Conversion Unit

- OCT Reactor
  - Operators need to be aware that desorption step of the activation/regeneration of OCT Reactor can take as long as 72 hrs
    - Target of the step is to achieve a delta concentration of moisture between the reactor offgas and the feed of less than 1 ppmv
      - Need to measure moisture in N2 leaving reactor AND
      - In N2 entering the reactor
      - If moisture in N2 entering reactor is high → find out reason!



Olefins Conversion Unit

- OCT Reactor Feed Heater
  - Time delay for LL flow interlock in OCT Reactor Feed Heater too short
  - LL pressure trip of heater burners did not match liberation curves
  - Range of duty controllers in heaters too small
  - Ramping up temperatures in heaters too fast during refractory dryout
  - Operating heaters with too low/high excess O2

Olefins Conversion Unit

- Deethylenizer
  - Overpressure of tower caused by N_2 accumulation, vent valve not sized to deal with excess N_2 (from padding)
  - Over-reboiling of tower, too tight ethylene spec in tower bttms
  - Under-reboiling of tower, ethylene leak to Depropylenizer
  - Over-flashing of ethylene in Deethylenizer reflux drum when running pump on min flow for long periods of time
  - Fresh ethylene pump frequent trips
  - Not following proper dryout procedure for OCU, wasting very large quantities of N_2 and time
  - Propylene BL pressure too low to be able to inventory propylene into towers during operation



Olefins Conversion Unit

- Deethylenizer
  - Plugging of C_3R strainers due to poor commissioning of lines (dust, debris accumulation)
  - Checking of tower leaks during pressuring of tower
  - Inventorying wrong blend of C2/C3/C4 prior to starting up
  - Inventorying liquid propane in Deethylenizer at low pressure
  - High propylene concentration in tower ovhd (high temperature), which gets recycled back to the OCT Reactor and limits equilibrium conversion

- Deethylenizer
  - Pilot-operated PSV located in inlet of OCT Reactor/Feed Effluent Exchanger (discharges to downstream Deethylenizer) should be purchased with a back-flow preventer
    - Prevents accidental reverse flow through safety valve
      - This could happen if Deethylenizer is inventoried / commissioned on total reflux while the OCU loop sits at low pressure



D. Backflow Preventer:

- Prevents accidental reverse flow through safety valve.

This option, sometimes called a 'vacuum block,' prevents a pilot operated safety valve from reverse flow, when sufficient vacuum is present at the inlet flange. The backflow preventer also prevents reverse flow when the pressure at the outlet flange (superimposed back pressure) is greater than the current system pressure. Reverse flow will occur with any standard type or design of pilot operated safety valve, when sufficient reverse differential pressure exists. Reverse flow, should it be induced by a reverse differential pressure, will be prevented by this option.



Olefins Conversion Unit

- Depropylenizer
  - High concentrations of propylene in C_4 recycle, not keeping the right heat balance in the tower
  - Damaging the reflux pumps after running with too low level in the reflux drum
  - Leaking tubes in condenser:
    - Caused by operation at higher-than-design temperature, high concentration of C4’s in tower overhead
    - Corrosion of tubes due to lower-than-design CW flow, low velocities

Olefins Conversion Unit

- Depropylenizer
  - Problems controlling the Depropylenizer pressure → low CW temperature (design = 33°C, actual = 24°C), high instability introduced by hot-vapor bypass operation, tuning problems
    - Due to the low pressure, it was impossible to sent the bottoms to the Ethylene plant → not enough differential pressure. Bottoms had to be flared
    - The low pressure problem was temporarily solved by throttling the CW (down to 8% opening) and fully closing the hot-vapor bypass
  - High isobutene in OCU feed, from feed treatment (design = 2%, actual = 8 – 10%). Isobutene is a low boiler, thus it has a higher tendency to end up in the DeC3- ovhd



Olefins Conversion Unit

- Other
  - Poor adjustment of valves' stroke speed (solenoid vent valve diaphragm)
  - Fabricating additional spool-pieces to speed up maintenance group response
  - Leaking valves
  - Waiting to torque bolts after unit is in operation (hot torquing)
  - Welding of sliding supports of pipes operating at high temperature – pipe cannot grow! Also, not removing min stops of supporting springs
  - Poor insulation of lines

Olefins Conversion Unit

- Feed quality
  - Problem: poor performance of online analyzers, continuous fouling of analyzer lines
  - Cause: large quantity of green oil in the feed from OSBL (much higher than design)
  - Short-term solution: modify sample conditioning systems to allow purge of green oil
  - Long-term solution: impact of green oil on adsorbents, catalyst and equipment is unknown. Accelerated fouling may occur → need to understand source of green oil in order to make adjustments to upstream process

Olefins Conversion Unit

- OCU loop cleaning:
  - It is standard procedure to carry out loop cleaning of the OCU loop before startup
    - C4’s are circulated at high rate through filtering element (or reduced pump strainers) in order to remove construction debris and substances that could potentially poison the OCU catalyst
    - It is very important to dump (deinventory) the C4’s used for loop cleaning to OSBL – NOT to the feed tank!

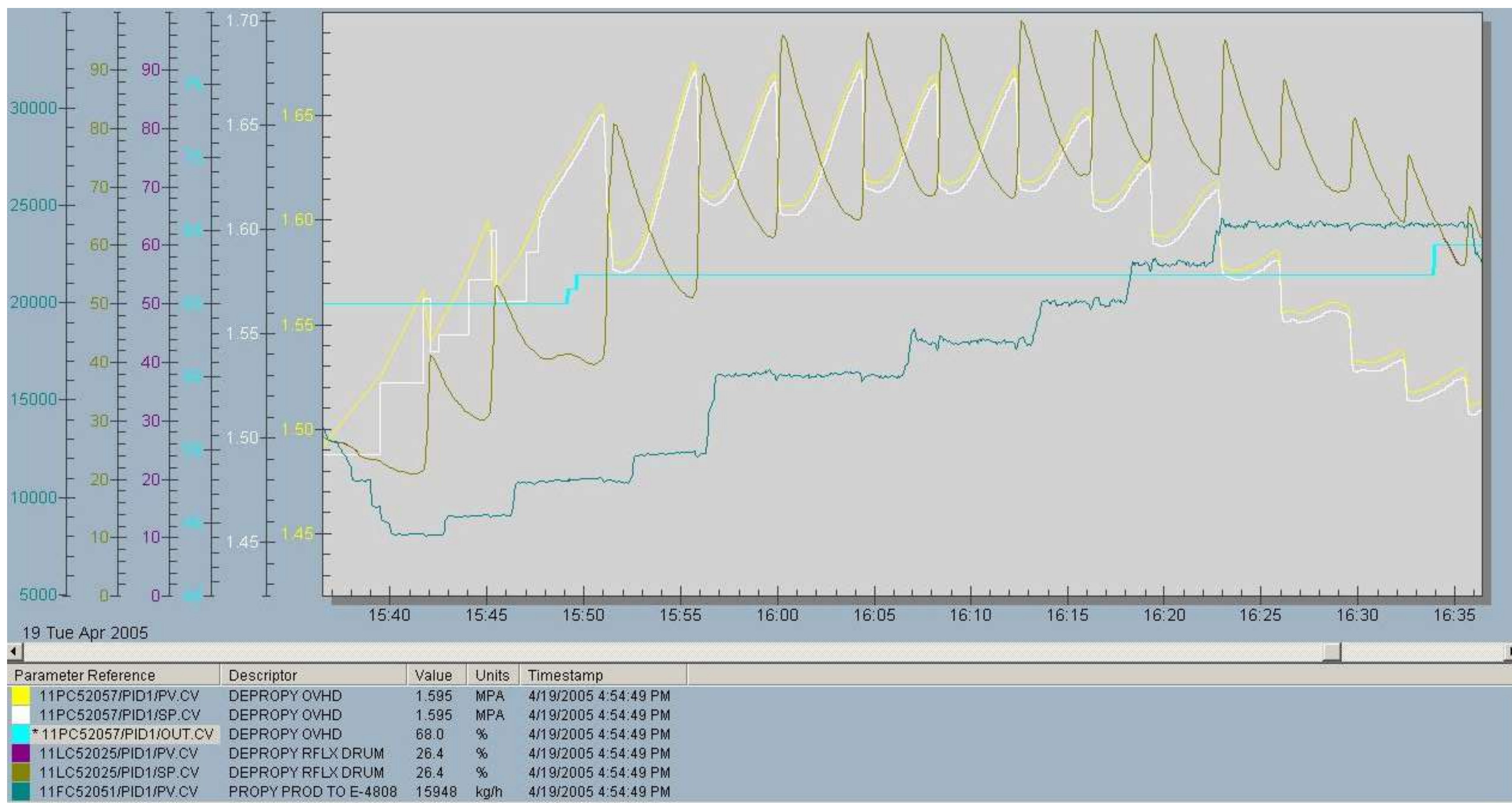


Olefins Conversion Unit

- Other
  - HH E/B ratio interlock based on volumetric flows, actual measurement based on mass flows
  - Electric heater not working properly
    - Problems drying out heater
  - No protection of N_2 lines due to overpressure (although provision was added in design)
  - Performing water-run of pumps without checking design pressure of pipes

- Hot Vapor Bypass
  - Difficulty in controlling Depropylenizer pressure due to low CW temperature:
    - Design CW temp = 33°C, actual = 24°C
    - Design tower pres = 1.78 MPaG, actual = 1.4 – 1.5 MPaG
  - Because of the low pressure, it was very difficult to send the bottoms to OSBL (no pump was provided). In addition, at lower pressure concentration of C4's in ovhd is higher → propylene off-spec
  - Using the hot bypass valve would introduce instability into the system: pressure would rise slowly and then it would drop very rapidly, due to vapor collapse

- Hot Vapor Bypass



- Hot Vapor Bypass
  - Depropylenizer overhead lines, hot bypass line and reflux drum not insulated → made system very sensitive to weather changes (day – night, rain, etc.)



- Low Cooling Water Velocity
  - Corrosion in Depropylenizer condenser caused by high operating temperatures and low CW velocity:



- Low Cooling Water Velocity
  - OCU CW system shared with cracker
  - Low CW velocity caused accelerated fouling of OCU exchangers, and was major cause for tube failure of Depropylenizer condenser
  - Although there was enough CW capacity in the plant, optimization of CW distribution was not done. Measurement of actual velocities in OCU should have been done
  - Large quantities of debris found inside exchanger. Proper commissioning of lines is very important

Engineering

- Ensure design adequately accounts for heat losses in regeneration systems
  - Provide for heat losses in piping in equipment design
  - Specify heat losses assumed and advise DEC to confirm
    - Heater datasheet notes
    - Instructions to DEC
  - Specification of screen size for OCT reactors
  - Additional analysis point in Deethylenizer reboiler inlet would help during start-up when the tower is operating under total reflux

Construction

- Ensure insulation work is completed before start-up
  - Check completeness of insulation work (flanges, shoes, etc)
  - Tighten flanges and insulate after heating

- Problem: wrong location of temperature controllers
  - For systems where a TIC controls the mixing of cold/hot streams (e.g., Treater inlet regen), the location of the TIC is very important
    - In one plant the TIC was installed in the cold line, upstream the mixing point. When the TIC setpoint was increased, the process value did not increase, which resulted in the output going to 100% which fully opened the hot valve
    - Make sure constructed plant reflects correct location of instrumentation as in P&ID!

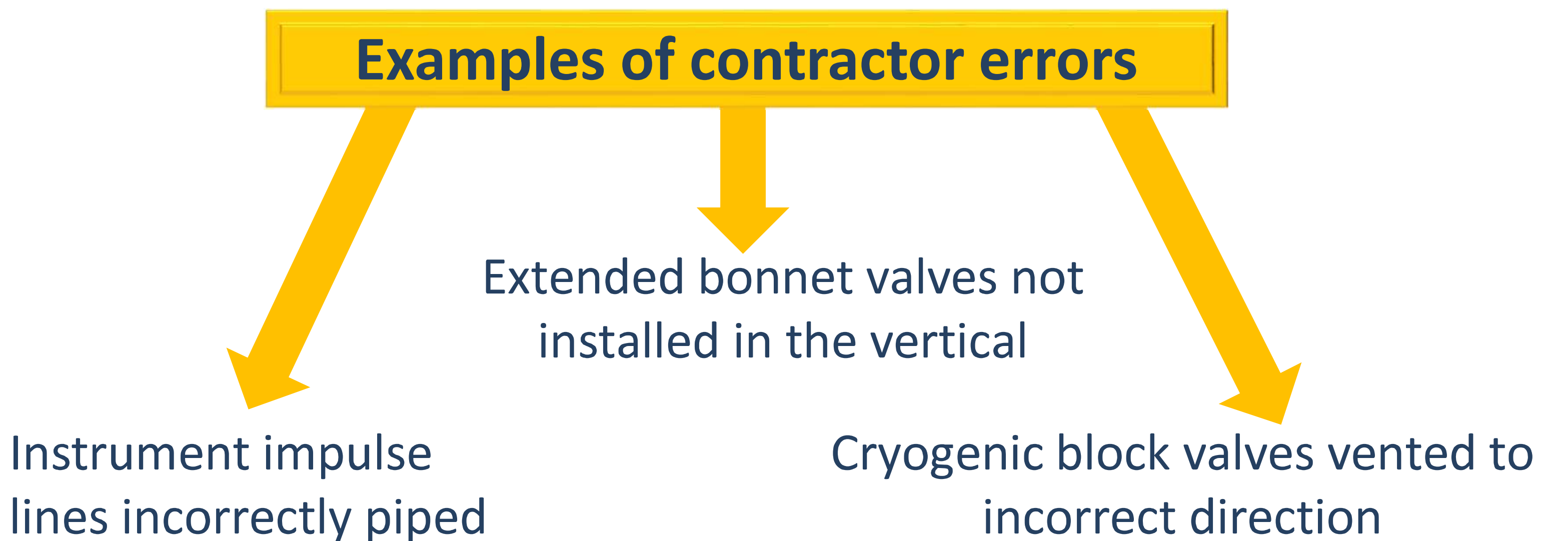
Construction

- Steam hammering:
  - This is an issue in most plants
  - Should avoid mixing sub-cooled condensate with 2-phase condensate
    - Sub-cooled condensate should have a dedicated line running back to the condensate drum

- Avoid pockets in regeneration return lines (which may carry water), and return lines for preload system

Construction

- Cryogenic Installations
  - Recent plant construction have had incorrect cryogenic installations
  - Required modifications prior to start-up, causing delays

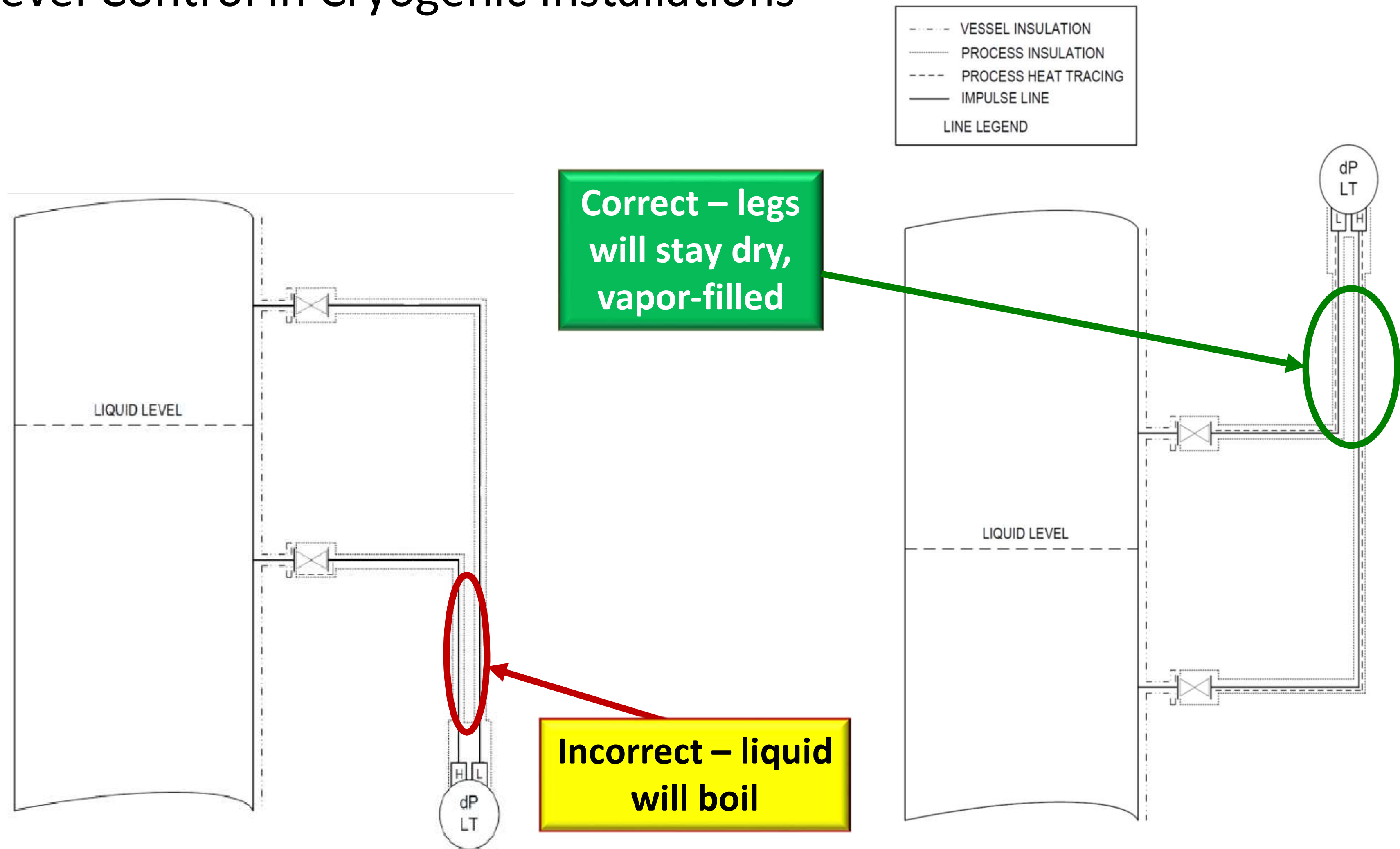


Construction

- Consequences of Incorrect Piping
  - When instrument impulse lines are piped like a standard liquid:
    - Liquefied gases boil in impulse lines around flow and level meters
    - Boiling liquid keeps changing density, causing erratic measurement
    - Unable to control flow or level; for example:
      - Deethylenizer reflux rate
      - Deethylenizer condenser refrigerant level

Contractors often miss this so Lummus now provides details in the PDP

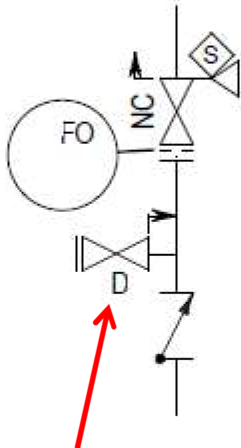
Level Control in Cryogenic Installations

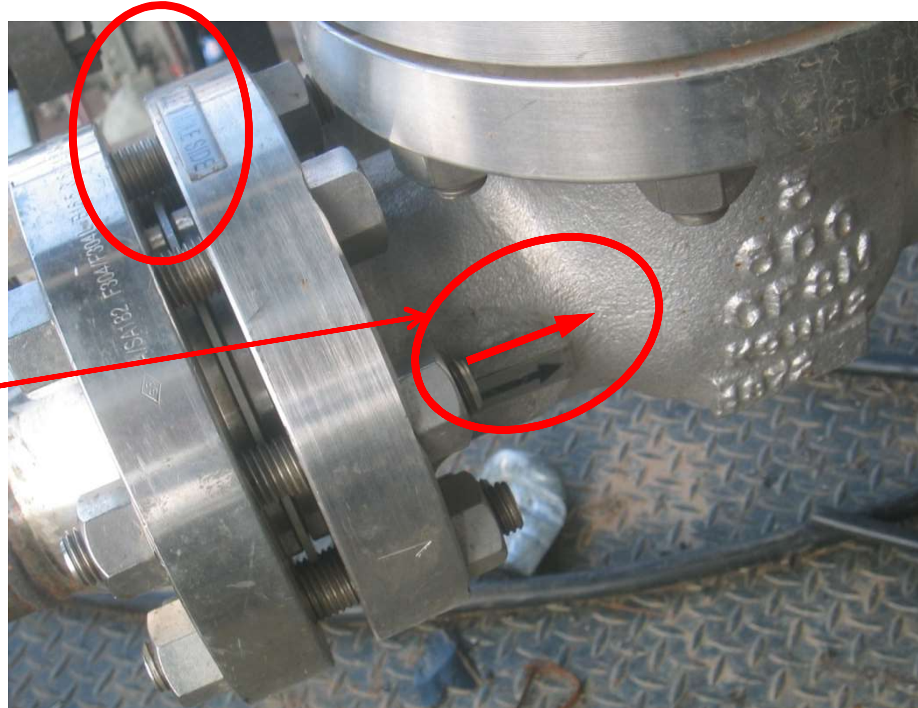


- Block Valves in Cryogenic Service

Valves vented to incorrect direction

- **Manufactures are all different!**





PID shows direction valve should vent BUT this valve shows what side the vent hole is on!

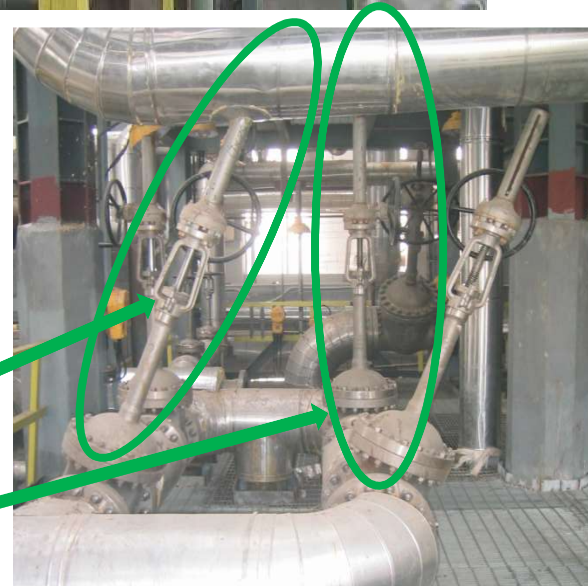
Arrow on valve from this manufacturer will be opposite of PID

- Block Valves in Cryogenic Service



Incorrect

Extended bonnets should be upright not horizontal or downward

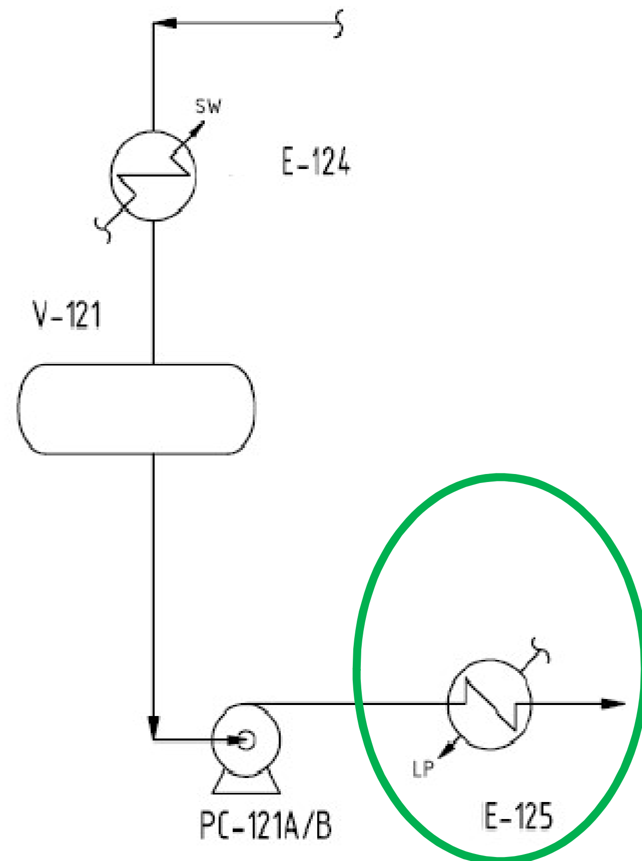


Correct

CBI

Construction

- Butene Stripping Vaporizer
  - For warming of treaters during regeneration some plants have a butene vaporizer
    - Recovers C_4s entrained in adsorbent pores
    - Stabilize return composition of regen gas to FG system
  - Several butene vaporizers have not functioned as required
    - Can not achieve required duty and temperature



Construction

- Butene Stripping Vaporizer

	E-125	E-613	New E-125
Type	V-BEU	H-BEU	V-BEM
Size mm	740 x 6096	700 x 6096	650 x 6096
Tube Side	C4 vaporizing	LP Steam	LP Steam
Shell Side	LP Steam	C_4 Vaporizing	C_4 Vaporizing
Area m^2	124	107	170
Vaporizing Duty	~50%	100%	100%

Unable to deliver duty

Smaller, horizontal exchanger was able to meet E-125 duty!

New exchanger with shell side boiling

HTRI-XIST indicated E-125 would work for full duty

Construction

- Butene Stripping Vaporizer
- Lummus worked with HTRI to find the problem
  - HTRI-XIST did not provide a very good analysis in the boiling zone with a large variation in the makeup of the shell side heat transfer coefficient giving unreliable results
  - HTRI-XIST predicts film boiling with the calculated heat flux is greater than critical
  - Film boiling is an unpredictable phenomena and it is recommended to design away from that flow regime
- Lummus specifies a horizontal, shell side boiling exchanger

Commissioning

- Plugging of suction strainer in C_3R compressor in Ethylene Plant
  - This was caused by debris carryover from one of the C3R users in the C4 complex shortly after commissioning of the lines
  - It is important to conduct proper commissioning (line blow) of refrigeration lines
  - Decision was made to install additional strainers in C_3R header returning from C4 complex to cracker
  - Installation of strainers took more than two weeks



Commissioning

- Ensure system is clean before start-up
  - OCU loop start-up procedures modified to include “loop cleaning” circulation to remove impurities from system prior to start-up – clean and flush with C_4’s



Commissioning

- Other
  - Debris in exchangers
  - Inadequate degreasing of strainers

- Electric Heater Dryout
  - Electrical heaters often installed in treater regeneration system to provide high temperature
  - Electrical heater brought into service early in the startup
  - Often the electrical heater is not adequately dried-out
    - Not be able to deliver full duty or can short-out



Commissioning

- Electric Heater Dryout
  - Each vendor's electrical heater has different dryout procedures
    - Contractors sometimes dry for too short a period or too quickly
    - Can cause a couple weeks delay in initial start-up



Lummus highlights this issue in Operating Manual

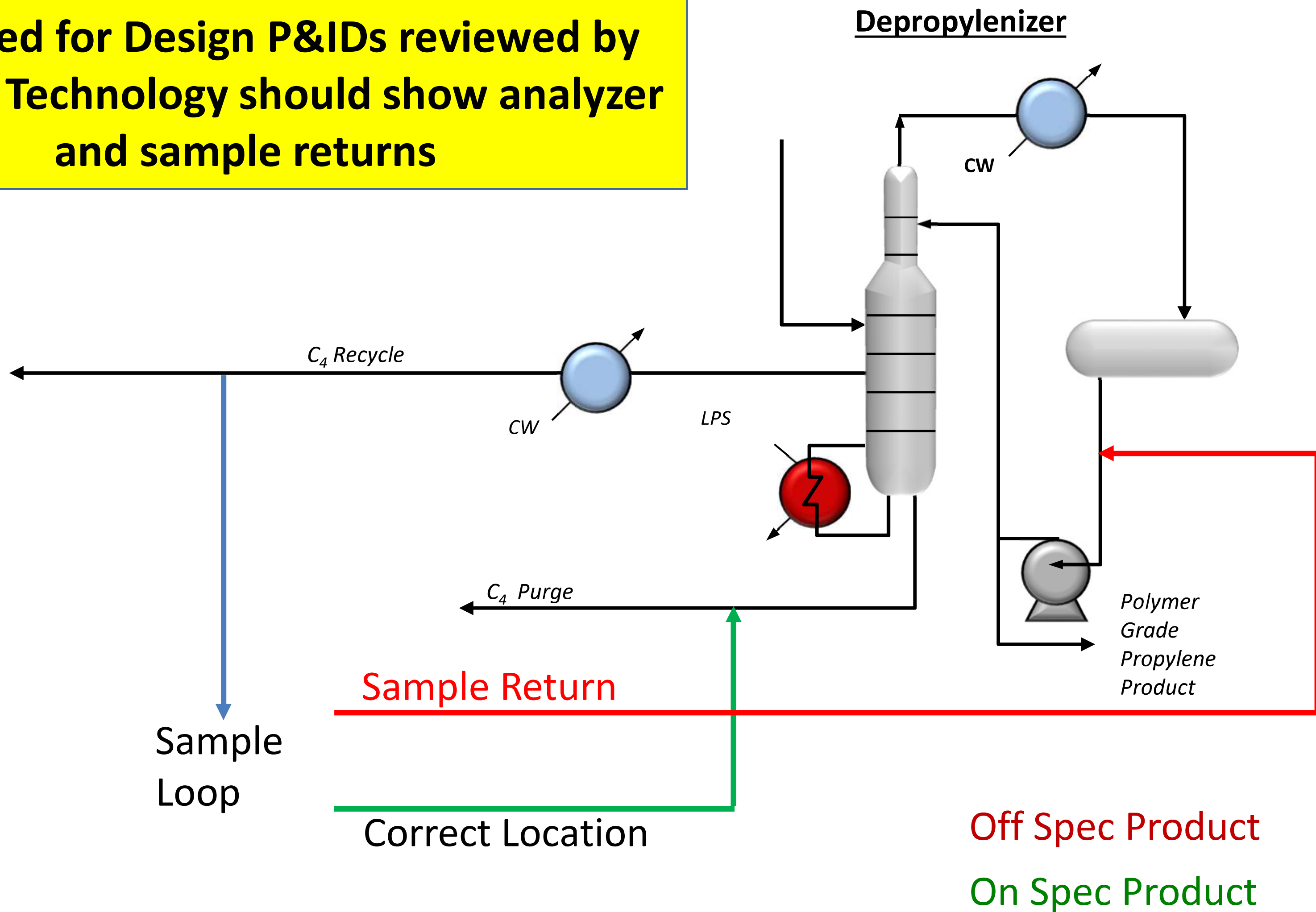
Laboratory

- Analytical
  - Testing of feed streams was not always conducted before initial feed-in → catalyst poisoning
  - Improper installation of OCT Reactor feed analyzer
  - Installation of wrong sampling station for OCT Reactor feed and effluent streams
  - Online analyzer does not analyze for n-butenes in Depropylenizer purge
  - Using the same sampling system for taking samples with very different composition (sample contamination)
  - Problems with Wobbe analyzers
  - Sometimes lab analysis methods are not clear even for the lab facility

Laboratory

- Analyzer/Sample Piping
  - Analyzer loops and sample stations
    - During detailed engineering, analyzer and sample loops and/or returns to process not correctly designed by the contractor
    - Some plants waste a substantial amount of feed or cross-contaminate systems leading to off-specification product quality

Approved for Design P&IDs reviewed by Lummus Technology should show analyzer and sample returns



Operations

- Operations Problems
  - Operating procedures were not available to the operators in the control room
  - Reading the SOM and detailed operating instructions is important – however, having a team discussion about a given activity just before the activity is carried out is more important
  - It is essential to inform and coordinate with other units (ethylene plant, BEU, etc.) when an activity is about to be carried out
  - Poor understanding of DCS and sequence controllers
  - Bypassing interlocks without doing the proper MOC (Management of Change) paperwork, and not keeping records of what has been bypassed/changed

Operations

- Operations Problems
  - Lack of supervision – there should be an experienced shift supervisor in the control room at all times
  - Operations and process engineers play an important role in any startup. However, the actual operation of the plant should be executed by qualified operators – it is not the job of the process engineer to manipulate the panel


CB&I